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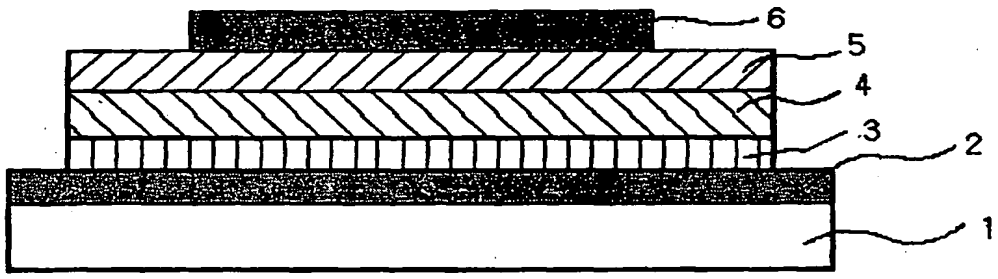
(54) Title: ELECTROLUMINESCENT DEVICES EMPLOYING ORGANIC LUMINESCENT MATERIAL/CLAY NANOCOMPOSITES



(57) Abstract: The present invention relates to an organic luminescent material/clay nanocomposite with improved luminescent efficiency and stability, which is prepared by blending an organic luminescent material with a nanoclay, and an electroluminescent device employing the same. The electroluminescent device of the invention comprises: a transparent substrate; a semitransparent electrode deposited on the transparent substrate; a clay nanocomposite emissive layer spin-coated with an organic EL material/clay nanocomposite, positioned on the semitransparent electrode; and, a metal electrode deposited on the clay nanocomposite emissive layer. Since the electroluminescent device of the invention provides improved luminescent efficiency and stability, it can be practically applied to the development of organic semi-conductor.

WO 01/72925 A1

ELECTROLUMINESCENT DEVICES EMPLOYING ORGANIC LUMINESCENT MATERIAL/CLAY NANOCOMPOSITES

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates to electroluminescent devices employing an organic luminescent material/clay nanocomposite, more specifically, to an organic luminescent material/clay nanocomposite with improved luminescent efficiency and stability, which is prepared by blending an organic luminescent material and a nanoclay, and electroluminescent devices employing the same.

Description of the Prior Art

The development of electroluminescent("EL") devices that emit light by applying an electric field has been continued, and polymer EL devices employing organic polymer/inorganic material nanocomposites have been developed and practiced in the art. These polymer EL devices utilize semiconductive inorganic materials, such as ZnS and CdS, and insulating inorganic materials such as SiO_2 and TiO_2. W. Que teaches luminescent polymers employing the semiconductive inorganic material, ZnS, and H. Mattoussi also teaches luminescent polymers employing the semiconductive inorganic material, CdS(see: W. Que, Applied Physics Letter, 73:2727, 1998; H. Mattoussi, Journal of Applied Physics, 83:796, 1998). Besides, luminescent polymers employing an insulating inorganic material of SiO_2 have been reported by S. A. Carter, and luminescent polymers employing an insulating inorganic material of TiO_2 have been reported by L. Gozano(see: S. A. Carter, Applied Physics Letters, 71:1145, 1997; L. Gozano, Applied Physics Letters, 73:3911, 1998), in which the inorganic materials

mixed with polymers have been used as luminescent layers. The inorganic nanomaterials are known to help charge transport. However, the luminescent efficiencies of these newly developed EL devices are not considerably improved compared to the luminescent efficiencies of the conventional devices, and the stability of the devices are low due to the unsolved problem of penetration of oxygen and water.

Therefore, there are strong reasons for developing and exploring a novel material that can solve the problems associated with the polymer EL devices described above.

SUMMARY OF THE INVENTION

The present inventors made an effort to provide a material that can improve the luminescent efficiency and stability of EL devices, and discovered that EL devices employing a luminescent material, an organic EL material/clay nanocomposite, prepared by blending an organic EL materials and a nanoclay, show the improved luminescent efficiency and stability.

A primary object of the present invention is, therefore, to provide an organic EL material/clay nanocomposite, prepared by blending an organic EL material and a nanoclay.

The other object of the present invention is to provide EL devices employing the organic EL material/clay nanocomposite.

BRIEF DESCRIPTION OF THE DRAWINGS

The above, the other objects and features of the invention will become apparent from the following descriptions given in conjunction with the accompanying drawings, in which:

Figure 1 is a schematic diagram depicting the structure of a nanoclay.

Figure 2 is a cross-sectional view of an EL device of the present invention employing an organic EL material/clay nanocomposite.

Figure 3 is a graph showing the photoluminescence intensity versus time of an EL device of the present invention.

Figure 4 is a graph showing the quantum efficiencies of EL devices with different nanoclay contents.

<Explanation of major parts of the drawings>

1: transparent substrate
2: semitransparent electrode
3: hole transporting layer
4: clay nanocomposite emitting layer
5: electron transporting layer
6: metal electrode

DETAILED DESCRIPTION OF THE INVENTION

Organic EL material/clay nanocomposite of the present invention, in which an organic EL material and a nanoclay are blended, is prepared in a form of quantum well resulting from the intercalation of the organic EL material into the nanoclay. The nanoclay used is a laminated inorganic material with 0.2 to 2 nm thickness in the vertical direction and 10 to 5,000 nm width in the horizontal direction, and the organic EL material is intercalated between the layered plates(see: Figure 1). The nanoclay includes materials having an insulating property, preferably montmorillonite (MMT), laponite and kaolinite. The 2-dimensional plate structure blocks electron or hole transport so that electric charges are

collected between the plates, resulting in the improvement of the electron-hole recombination probability or the EL efficiency; and, it also considerably decreases the penetration of oxygen and moisture, which, in turn, improves the stability of the organic EL material/clay nanocomposite(see: Figure 3).

The organic luminescent materials employed in the organic EL material/clay nanocomposite of the present invention can be used for many different types of organic luminescent materials including, but not limited to, emissive conjugated polymers, emissive non-conjugated polymers, copolymers of conjugated and nonconjugated segments, blends of the emissive polymer with emissive or non-emissive polymers, emissive small organic molecules such as monomers or oligomers, blends of the small organic molecules with emissive or non-emissive polymers, or blends of emissive small organic molecules and non-emissive small organic molecules.

Suitable choices for the emissive conjugated polymers include, but not limited to: poly(*p*-phenylene vinylene) and its derivatives such as MEH-PPV(poly[2-methoxy-5-(2'-ethylhexyloxy)-*p*-phenylene vinylene]), poly(pyridyl vinylene phenylene vinylene)(PPyVPV), and poly[1,4-(2,5-bis(1,4,7,10-tetraoxaundecyl))phenylene vinylene]; polythiophene and its derivatives such as poly[3-hexylthiophene-*co*-3-cyclohexylthiophene] and poly[3-(4-methoxypheyl)thiophene-2,5-diyl], poly(*p*-phenylene) and its derivatives such as dimethoxy-poly(*p*-phenylene), ladder poly(dihydrophenanthrene), and ladder poly(1,4-phenylene-2,5-thiophene); polyfluorene and its derivatives such as poly(9,9-dioctylfluorene), poly(2,7-bis(p-styryl)-9,9'-di-n-hexylfluorene sebacate); poly(arylene vinylene), where the arylene may be such moieties as naphthalene, anthracene, furylene, thienylene, oxadizole, and the like, or one of said moieties with functionalized substituents at various positions; derivatives of poly(arylene vinylene), where the arylene may be as in above, substituted at various

positions on the arylene moieties; polyarylene and their derivatives substituted at various positions on the arylene moiety; polypyrrole and its derivatives; polyquinoline and its derivatives; polyacetylene and its derivatives; and, polyaniline and its derivatives, and the like.

The emissive non-conjugated polymers have non-conjugated main chains and side chains substituted with emissive functional groups such as anthracene.

Organic luminescent monomers or oligomers include metal chelate complexes of ligand structure such as luminescent alumina quinone(Alq3), and rubrene, anthracene, perylene, coumarin 6, Nile red, aromatic diamine, TPD(N,N'-diphenyl-n,n'-bis-(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine), TAZ(3-(4-biphenyl)-4-phenyl—(4-tert-butylphenyl)1,2,4-triazole), and DCM(dicyanomethylene)-2-methyl-6-(p-dimethylaminostyryl)-4*H*-pyran), or derivatives thereof.

Non-emissive polymers, such as poly(*m*-methylacrylic acid), polystyrene, and poly(9-vinylcarbazole) can be used as a matrix for blends with emissive compounds; besides, blends of organic luminescent monomers or oligomers with the above emissive conjugated polymers or emissive non-conjugated polymers can be employed for the nanocomposite with an clay.

EL devices of the present invention employing organic EL material/clay nanocomposite include a substrate which is composed of a semitransparent electrode, a hole-transporting (or injecting) layer, an organic EL material/clay nanocomposite, an electron-transporting (or injecting) layer, and a metal electrode.

The EL devices of the invention have charge-injecting contact layers which serve as electrodes for applying the voltage across the thin layer. The devices of the invention have a semi-transparent conducting layer coated on a transparent substrate, which is utilized as a charge-injecting contact layer. The conducting layer includes metal oxides such as lead oxide, ITO(indium tin oxide),

doped conducting polymers such as doped polyaniline, doped polypyrrole, PEDOT(polyethylene dioxyhiophene) or doped polythiophene and the like. The transparent substrates may be rigid or mechanically flexible, which include glass, quartz, plastics such as polyethylene terephthalate, and the like; and, the metal electrode includes aluminum, magnesium, lithium, calcium, copper, silver, iron, platinum, indium, palladium, tungsten, zinc, gold, lead and alloys thereof, which were also employed for an charge-injecting contact layer. Besides, graphite, inorganic semiconducting semiconductors such as silicon, germanium, gallium arsenide, silicon, and the like can be employed for an charge injecting electrode.

A preferred embodiment of the EL device of the present invention employing an organic EL material/clay nanocomposite is illustrated in Figure 2. As shown in Figure 2, the EL device employing an organic EL material/clay nanocomposite comprises a transparent substrate(1), such as glass, a semitransparent electrode(2) deposited on the transparent substrate, a clay nanocomposite emissive layer(4) spin-coated with the organic EL material/clay nanocomposite of the invention, positioned on the semitransparent electrode, and a metal electrode(6) deposited on the clay nanocomposite emissive layer. To improve the EL efficiency, the device may be further provided with a hole transporting (or injecting) layer(3) between the semitransparent electrode(2) and the clay nanocomposite emissive layer(4) and/or an electron transporting (or injecting) layer(5) between the clay nanocomposite emissive layer(4) and the metal electrode(6), where the hole transporting layer is preferably made of one of polymers with hole-transporting moiety including poly(9-vinylcarbazole) and its derivatives; small organic materials(monomers or oligomers) including 4,4'-dicarbazolyl-1,1'-biphenyl(CBP), TPD(N,N'-diphenyl-N,N'-bis-(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine), NPB(4,4'-bis[N-(-naphthyl-1-)-N-phenyl-amino]-biphenyl),

triethylamine, pyrazole, and their derivatives; or, other organic materials(monomers, oligomers, and polymers) containing hole transporting moieties. The electron transporting layer is preferably made of TPBI(2,2',2'-(1,3,5-benzenetriyl)tris[1-phebenzimidazole]), poly(phenyl quinoxaline), 1,3,5-tris[(6,7-dimethyl-3-phenyl)quinoxaline-2-yl]benzene(Me-TPQ), polyquinoline, tris(8-hydroxyquioline)aluminum(Alq3), {6-N,N-diethylamino-1-methyl-3-phenyl-1H-pyrazole[3,4-b]quinoline} (PAQ-Net2), or other organic materials(monomers, oligomers, and polymers) containing electron transporting moieties.

The luminescent efficiency of EL devices is expressed in terms of external quantum efficiency, e.g., percentage(%) of the number of externally emitted photons against the number of injected electrons. For the EL devices of the present invention, as the content of nanoclay is increased, the higher external quantum efficiency is obtained and the gas penetration is decreased, resulting in the distinctive decrease of photoluminescent decay caused by protecting the penetration of the external gases such as oxygen and moisture. Therefore, the luminescent efficiency and the stability of the EL devices are simultaneously improved as the content of nanoclay is increased.

The present invention is further illustrated by the following examples, which should not be taken to limit the scope of the invention.

Example 1: Measurement of the luminescent intensity of EL device

An emissive material, MEH-PPV(poly[2-methoxy-5-(2'-ethyl-hexyl)-*p*-phenylene vinylene]) was dissolved in 1,2-dichloroethane, and a nanoclay, montmorillonite(MMT) was blended with the MEH-PPV solution in the ratio of 1:1(w/w). The resultant was sonicated to intercalate the MEH-PPV

chain into MMT. And then, an EL device was prepared by spin-coating the mixture onto an ITO glass in 150 nm thickness to give a thin film, followed by thermal evaporation of an Al electrode deposited on the emissive nanocomposite layer. The EL intensity with the function of time was measured using a photodiode(Newport 818-UV) connected to optical powermeter(Newport 1830-C) after applying a short wavelength light of 340nm.

Comparative Example 1: Measurement of the luminescent intensity of EL device not employing nanoclay

An EL device was prepared in a similar manner as in Example 1, except that nanoclay was not employed. The EL intensity with the function of time was measured, and compared to the result of Example 1(see: Figure 3). Figure 3 is a graph showing the time course of the effect of nanoclay on the photoluminescence, where (○) and (□) represents the results of Example 1 and Comparative Example 1. As shown in Figure 3, the photoluminescent intensity of the nanocomposite EL material employing nanaoclay decreased relatively slower than that of the EL material without nanaoclay, demonstrating that the luminescent stability of the nancomposite EL material employing nanaoclay was substantially improved.

Example 2: Measurement of the external quantum efficiency of EL devices

EL devices not employing nanoclay were prepared in a similar manner as in Example 1, except that the blending ratio of MMT and MEH-PPV ranged from 1:2 to 1:5(w/w). The external quantum efficiencies of the EL devices prepared in Example 1, Comparative Example 1, and Example 2, depending on the electric current, were measured using Keithley 236 Source measurement unit(see: Figure 4). Figure 4 is a

graph showing the external quantum efficiencies of the EL devices, where (■) represents the change of the external quantum efficiency of an EL device not including MMT, prepared in Comparative Example 1, (●), an EL device in which the blending ratio of MMT and MEH-PPV is 1:5(w/w), (▲), an EL device in which the blending ratio of MMT and MEH-PPV is 1:2(w/w), and (▼), an EL device in which the blending ratio of MMT and MEH-PPV is 1:1(w/w), prepared in Example 1. As shown in Figure 4, the external quantum efficiency of an EL device in which the blending ratio of MMT and MEH-PPV is 1:1(w/w) showed the maximum value of 0.38%(photons/electrons), while, the external quantum efficiency of the EL device not containing MMT, prepared in Comparative Example 1, showed 0.004%(photons/electrons). Therefore, it was clearly demonstrated that the external quantum efficiency was dramatically increased when the nanoclay was blended with the emissive materials.

Example 3: Preparation of EL device(I)

The material prepared by blending an EL material, poly(xylylidene tetrahydrothiophenium)(PTHT) and a nanoclay, MMT in the ratio of 1:1(w/w) was spin-coated on the ITO plate in 150nm thickness and heated at 170℃ for 3 hours to give a clay nanocomposite emissive layer. And then, an aluminum electrode was deposited in 100 nm thickness and at a deposition rate of 5Å/s on the resulting material by the aid of a thermal evaporator to give an EL device.

Example 4: Preparation of EL device(II)

An EL device was prepared in a similar manner as in Example 3, except that MEH-PPV(poly[2-methoxy-5-(2'-ethyl-hexyloxy)-*p*-phenylene vinylene]) was used as an EL material, and spin-coating of the material was followed to form an 120 nm-thick film, and then a calcium electrode was deposited thereon.

Example 5: Preparation of EL device(III)

An EL device was prepared in a similar manner as in Example 2, except that the material prepared by doping poly(9-vinylcarbazole) with alumina quinone(Alq3) in 5% weight ratio was used as an EL material, and spin-coating was followed to form 120nm-thick film, and then a magnesium electrode was deposited.

Example 6: Preparation of EL device(IV)

A hole transporting material, poly(9-vinylcarbazole) (PVK) was spin-coated on ITO substrate and then a material prepared by blending an EL material, MEH-PPV and a nanoclay in the weight ratio of 1:1 was spin-coated on the top of the PVK film in 100nm thickness. And then, an electron transporting material, alumina quinone (Alq3) was deposited in 50nm thickness and at a deposition rate of 10Å/s using a thermal evaporator. On the resulting material, an aluminum electrode was deposited in 100nm thickness and at a deposition rate of 5Å/s using the same thermal evaporator to give an EL device.

As clearly described and demonstrated as above, the present invention provides an organic EL material/clay nanocomposite with improved luminescent efficiency and stability, which is prepared by blending an organic EL material with a nanoclay, and EL devices employing the same. The EL device of the invention comprises: a transparent substrate; a semitransparent electrode deposited on the transparent substrate; a clay nanocomposite emissive layer spin-coated with an organic EL material/clay nanocomposite, positioned on the semitransparent electrode; and, a metal electrode deposited on the clay nanocomposite emissive layer. Since the electroluminescent device of the invention provides improved luminescent efficiency and

stability, it can be practically applied to the development of organic semi-conductor.

Although the preferred embodiments of present invention have been disclosed for illustrative purpose, those who are skilled in the art will appreciate that various modifications, additions, and substitutions are possible, without departing from the spirit and scope of the invention as disclosed in the accompanying claims.

WHAT IS CLAIMED IS:

1. An organic luminescent material/clay nanocomposite prepared in a form of quantum well, by blending an organic luminescent material and a nanoclay.

2. The organic luminescent material/clay nanocomposite of claim 1, wherein the nanoclay is a laminated inorganic material with 0.2 to 2 nm thickness in the vertical direction and 10 to 5,000 nm width in the horizontal direction.

3. The organic luminescent material/clay nanocomposite of claim 1, wherein the nanoclay is montmorillonite(MMT), laponite or kaolinite.

4. The organic luminescent material/clay nanocomposite of claim 1, wherein the organic luminescent material is emissive conjugated polymer; emissive non-conjugated polymer; organic luminescent monomer or oligomer; blends of the said emissive polymers; or, blends of the said emissive polymer and non-emissive polymer.

5. The organic luminescent material/clay nanocomposite of claim 4, wherein the emissive conjugated polymer is poly(*p*-phenylene vinylene), polythiophene, poly(*p*-phenylene), polyfluorene, polyarylene, poly(arylene vinylene), polyquinoline, polypyrrole, polyaniline, polyacetylene or derivatives thereof.

6. The organic luminescent material/clay nanocomposite of claim 4, wherein the emissive non-conjugated polymer has non-conjugated main chains and side chains substituted with emissive functional groups.

7. The organic luminescent material/clay nanocomposite of claim 4, wherein the organic luminescent

monomer or oligomer is a metal chelate complex of ligand structure, rubrene, anthracene, perylene, coumarin 6, Nile red, aromatic diamine, TPD(N,N'-diphenyl-N,N'-bis-(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine), TAZ(3-(4-biphenyl)-4-phenyl-(4-tert-butylphenyl)1,2,4-triazole), DCM(dicyanomethylene)-2-methyl-6-(p-dimethylaminostyryl)-4H-pyran) or derivatives thereof.

8. The organic luminescent material/clay nanocomposite of claim 4, wherein the non-emissive polymer is poly(*m*-methylacrylic acid), polystyrene or poly(9-vinylcarbazole).

9. An electroluminescent(EL) device which comprises:

a transparent substrate;

a semitransparent electrode deposited on the transparent substrate;

a clay nanocomposite emissive layer spin-coated with the organic EL material/clay nanocomposite of claim 1, positioned on the semitransparent electrode; and,

a metal electrode deposited on the clay nanocomposite emissive layer.

10. The electroluminescent(EL) device of claim 9, wherein the transparent substrate is glass, quartz or transparent polyethylene terephthalate(PET).

11. The electroluminescent(EL) device of claim 9, wherein the semitransparent electrode is lead oxide, ITO(indium tin oxide), doped polyaniline, doped polypyrrole, PEDOT(polyethylene dioxythiophene) or doped polythiophene.

12. The electroluminescent(EL) device of claim 9, wherein the metal electrode is aluminum, magnesium, lithium, calcium, copper, silver, iron, platinum, indium, palladium, tungsten, zinc, gold, lead or an alloy thereof.

13. An electroluminescent(EL) device which comprises:

a transparent substrate;

a semitransparent electrode deposited on the transparent substrate;

a hole transporting layer positioned on the semitransparent electrode;

a clay nanocomposite emissive layer spin-coated with the organic EL material/clay nanocomposite of claim 1, positioned on the hole transporting layer; and,

a metal electrode deposited on the clay nanocomposite emissive layer.

14. The electroluminescent(EL) device of claim 13, wherein the hole transporting layer is made of polymer with hole-transporting moiety including poly(9-vinylcarbazole) and its derivatives; small organic material including 4,4'-dicarbazolyl-1,1'-biphenyl(CBP), TPD(N,N'-diphenyl-N,N'-bis-(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine), NPB(4,4'-bis[N-(-naphthyl-1-)-N-phenyl-amino]-biphenyl), triarylamine, pyrazole, and derivatives thereof; or, organic material containing a hole transporting moiety.

15. An electroluminescent(EL) device which comprises:

a transparent substrate;

a semitransparent electrode deposited on the transparent substrate;

a clay nanocomposite emissive layer spin-coated with the organic EL material/clay nanocomposite of claim 1, positioned on the semitransparent electrode;

an electron transporting layer positioned on the clay nanocomposite emissive layer; and,

a metal electrode deposited on the electron transporting layer.

16. The electroluminescent(EL) device of claim 15, wherein the electron transporting layer is TPBI(2,2',2'-(1,3,5-phenylene-tris[1-phenyl-1*H*-benzimidazole]),

poly(phenyl quinoxaline), 1,3,5-tris[(6,7-dimethyl-3-phenyl)quinoxaline-2-yl]benzene(Me-TPQ), polyquinoline, tris(8-hydroxyquioline)aluminum(Alq3), {6-N,N-diethylamino-1-methyl-3-phenyl-1H-pyrazolo[3,4-*b*]quinoline}(PAQ-NEt_2), or organic material containing an electron transporting moiety.

17. An electroluminescent(EL) device which comprises:

a transparent substrate;

a semitransparent electrode deposited on the transparent substrate;

a hole transporting layer of claim 14 positioned on the semitransparent electrode;

a clay nanocomposite emissive layer spin-coated with the organic EL material/clay nanocomposite of claim 1, positioned on the hole transporting layer;

an electron transporting layer of claim 16 on the clay nanocomposite emissive layer; and,

a metal electrode deposited on the electron transporting layer.

Fig. 1

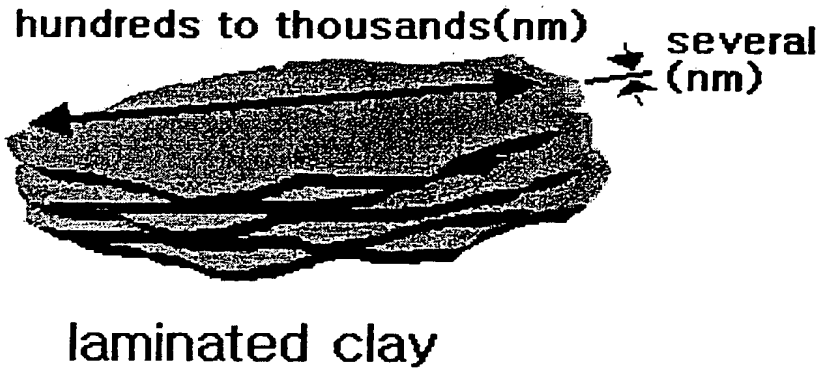


Fig. 2

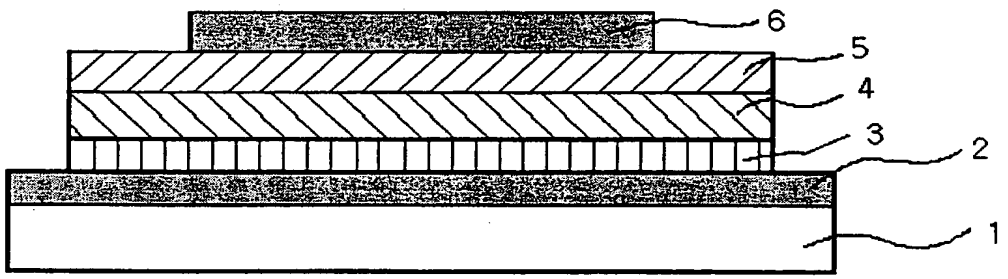


Fig. 3

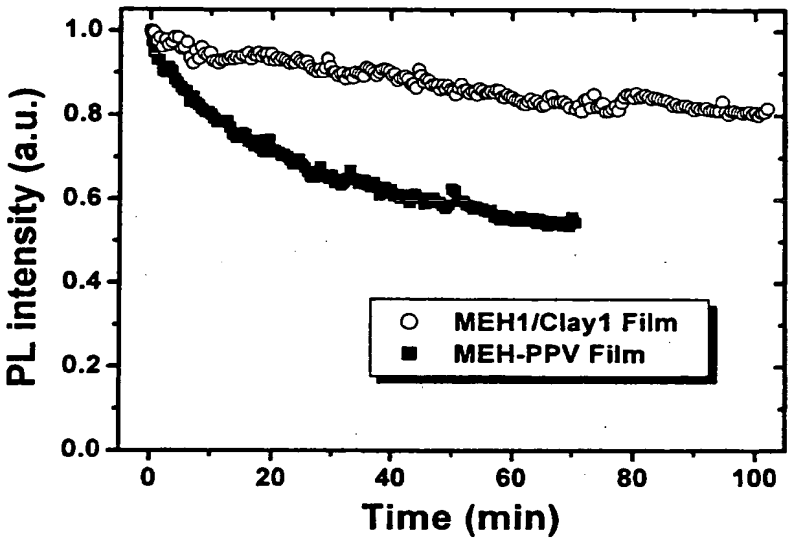
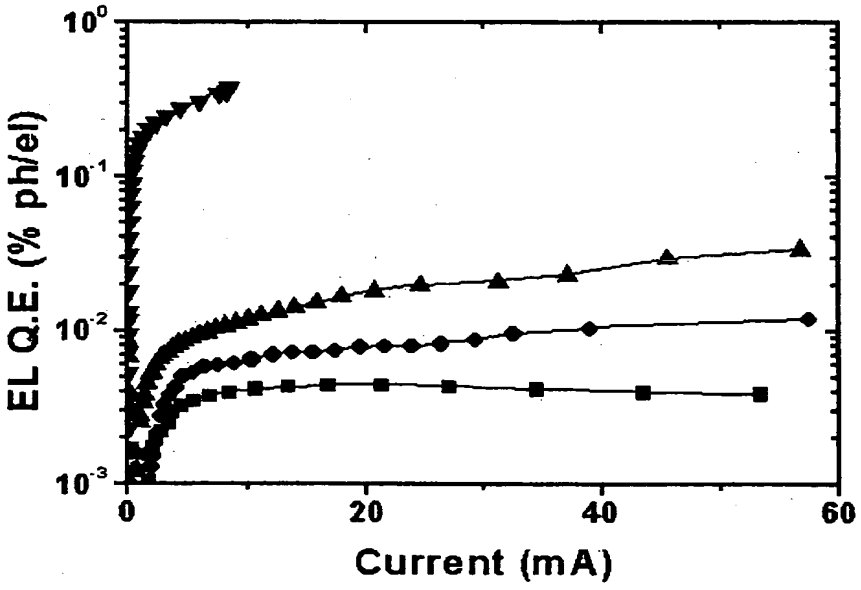


Fig. 4



INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR01/00534

A. CLASSIFICATION OF SUBJECT MATTER

IPC7 C09K 11/00, C09K 11/06, H05B 33/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimun documentation searched (classification system followed by classification symbols)

IPC7 C09K 11/06 , H05B 33/14, 33/26

Documentation searched other than minimun documentation to the extent that such documents are included in the fileds searched

Korean Patants and Applications for Inventions since 1975
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Electronic data base consulted during the international search (name of data base and, where practicable, search trerms used)

NPS, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5,653,914 A (CAMBRIDGE DISPLAY TECHNOLOGY LIMITED) 5 August 1997	1 - 12
A	JP 3-190991 A (TOSHIBA CORP) 20 August 1991	1
A	US 5,719,467 A (HEWLETT-PACKARD CO.) 17 February 1998	13 - 17

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Date of the actual completion of the international search	Date of mailing of the international search report
16 JULY 2001 (16.07.2001)	18 JULY 2001 (18.07.2001)

Name and mailing address of the ISA/KR	Authorized officer
Korean Intellectual Property Office Government Complex-Daejeon, Dunsan-dong, Seo-gu, Daejeon Metropolitan City 302-701, Republic of Korea	CHOI, Seung Keun
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
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